

Standard Operating Procedure

**for the USGS Reston Biogeochemical Processes in Groundwater Laboratory
(RBPGL)**

Determination of Non-volatile Dissolved Organic Carbon (NVDOC) in Water

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Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:

$$^{\circ}\text{F} = (1.8 \times ^{\circ}\text{C}) + 32$$

Temperature in degrees Fahrenheit (°F) may be converted to degrees Celsius (°C) as follows:

$$^{\circ}\text{C} = (^{\circ}\text{F} - 32) / 1.8$$

Concentrations of chemical constituents in water are given either in milligrams per liter (mg/L) or micrograms per liter (µg/L).

▮ Acronyms and Abbreviations

ASTM	American Society for Testing and Materials
CO ₂	Carbon dioxide
C	Carbon
CV	Coefficient Verification
gm	Gram
HC	Hydrocarbons
HCl	Hydrochloric acid, 37% Reagent grade
ID	Identification
C ₆ H ₄ -1,2-(CO ₂ K) ₂	Phthalic acid dipotassium salt
LIMS-NVDOC	Laboratory Information Management System for Non-volatile Dissolved Organic Carbon
mg/L	Milligram per liter
MDL	Method detection limit
Min	Minute
Milli-Q	Milli Q water
MSDS	Material Safety Data Sheets
MW	Molecular weight
N	Normality (equivalents per liter)
n	Number
NDIR	Non-dispersive Infrared
NVDOC	Non-volatile Dissolved Organic Carbon
P/N	Part number

ppm	Parts per million
%RSD	Percent Relative standard deviation
psi	Pounds per square inch
BPGL	Reston Biogeochemical Processes in Groundwater Laboratory
RSD	Relative standard deviation
SD	Standard deviation
SW	Surface water
S	Standard
SOP	Standard Operating Procedure
TC	Total Carbon
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon
USGS	U.S. Geological Survey
US EPA	U.S. Environmental Protection Agency
VFW	Volatile Free water
VOA	Volatile organic analysis

Symbols

Ω	Ohm
<	Less than
>	Greater than
~	Approximately
=	Equal
\geq	Greater than or equal to
\leq	Less than or equal to
\pm	Plus or minus
%	Percent
$^{\circ}\text{C}$	Degree Celsius

Determination of Non-volatile Dissolved Organic Carbon (NVDOC) in Water

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Summary of Procedure

This document describes the Standard Operating Procedure (SOP) for the determination of non-volatile dissolved organic carbon (NVDOC) of the U.S. Geological Survey Reston Biogeochemical Processes in Groundwater Laboratory (RBPGL), Reston Virginia (water.usgs.gov/nrp/biogeochemical-processes-in-groundwater). Water samples are analyzed for NVDOC by a combination high temperature combustion/non-dispersive infrared detector (NDIR) technique using a Shimadzu TOC-V csn Total Organic Carbon Analyzer with Control V System Software. Prior to analysis, filtered water samples are acidified with hydrochloric acid (HCl) to a pH less than 2. The acidified samples are sparged with a high purity carrier gas (Zero Air, 99% free of impurities) to remove the inorganic carbon. Each sample is injected directly into a combustion tube containing platinum oxidation catalyst that is housed in a high temperature furnace. The tube is heated to 680°C to vaporize the aqueous sample, reducing all carbon compounds to CO₂. The gas stream is then passed through a halogen scrubber to remove chlorides and then to a cooling humidifier (water trap) prior to the measurement of CO₂ in the detector. The resulting peak area is integrated by the Shimadzu Control V System Software, with concentrations of combustionally oxidizable nonvolatile organic carbon being proportional to the

concentration of carbon in the sample, (Shimadzu, 2001). The analyst can measure concentrations of NVDOC down to a detection limit of 0.08 mg/L of Carbon (C). A detailed description of the sampling methodology can be found at the USGS RBPGL web site (*water.usgs.gov/nrp/biogeochemical-processes-in-groundwater*, U.S. Geological Survey, 2014).

Operating Range

The typical analytical linear range of this method is 0.5-30 mg/L of C. Samples with higher concentrations must be diluted. Data are reported with two significant figures.

Reference Materials and Documentation

Reference Materials Used, Storage Requirements, and Stability

The RBPGL uses Potable WatR™ organic carbon referenced standards from ERA Laboratories, (<http://www.eraqc.com/Resources/ProductCatalogs>). Eight verified reference and calibration standards with concentrations between 3-13 mg/L of carbon are diluted with volatile-free water (VFW) for the Potable WatR™, and 2 N (Normality) HCl 37% Reagent Grade for the calibration standards. The standards are kept in refrigerated storage at 4 °C and remain stable for approximately 60 days.

Documentation

All calibration results are stored in the Reston Laboratory Information Management System for Non-Volatile Dissolved Organic Carbon Samples (LIMS-NVDOCS) associated with field id number, sample submission date, and project identifier. The requisite Microsoft Excel® data spreadsheet is created by the RBPGL analyst. Organic carbon certified reference material, blanks, and calibration standards are run with every batch of samples.

Sample Collection, Preparation, Analysis, Retention Time and Disposal

Field Sampling Containers, Collection Procedures, Preservation, and Handling Requirements

Water samples are collected in 20 -mL amber borosilicate glass precleaned EPA certified Volatile Organic Analysis vials (VOA) with open-top, septa-lined caps (septa are made of Teflon-lined silicone). This is a recommended size, however 40-mL borosilicate glass precleaned EPA certified VOA vials are acceptable. Vials with septa can be provided by RBPGL at a cost of \$3.00 each plus shipping to the sample submitter on request through e-mail or can be purchased at Scientific Specialties Service (www.scispec.com). The 20-mL P/N 276720-clear, P/N 276820-amber glass. The 40-mL P/N 276740-clear, 276840 –amber. Other scientific products retailer may also provide this product. For smaller sample volumes the submitter must first contact the RBPGL. The vials must be labelled clearly with a waterproof marker... All labels must list field id, initials of the sampler, date and time of sample collection, analyses to be done, and indicate the filter and preservative used. If duplicate samples are collected at one site they should be labelled “1 of 2” and “2 of 2”. Documentation accompanying the samples must indicate whether or not any samples contain a known or suspected hazard. If a hazard is noted, please provide a detailed description. If there is no known or suspected hazard, the documentation must include the word “None.” Samples to be analyzed by this method can be collected following sampling procedures outlined in the “U.S.G.S. National field manual, variously dated” available at <http://water.usgs.gov/owq/Fieldprocedures.html> . Samples are filtered through a 0.2 micron (μm) Gelman Supor™ filter (Pall-Gelman) and the vial is filled to the top of the shoulder, leaving enough space to add approximately 200 microliters (μL) (approximately four drops of HCl). Check that the sample pH is in the optimal range of 1.5-2.5; adjust pH with drop wise addition of HCl as necessary; (one drop \sim 50 μL). If sample volumes are less than 20-mL, add 10 μL of acid for every 1 mL of sample,

check pH. A minimum of 18-mL of water is required for sample analysis unless special arrangements are made in advance. Screw the cap onto the vial firmly, but do not overtighten cap. If the septa appears to be creased or if it is off-center, the vial may leak.

Place each vial into a foam sleeve, then into a Ziploc-type bag. Keep the vials chilled with wet ice or refrigerated until shipping. Keep all vials from the same site together. Secure the vials with additional packing materials and tape so they won't break during transport. Do not freeze the vials and do not ship the vials on blue ice, which can cause breakage.

Sample Shipping

Sample vials should be shipped on wet ice overnight, notifying RBPGL by e-mail that the samples having been shipped. The coolers must be sent Monday-Thursday by overnight delivery. If samples cannot be shipped on the day of collection, they should be kept chilled. Label the inside lid of the cooler with return address and telephone number using a waterproof marker. Coolers will be returned by RBPGL to the sample submitter at the submitter's expense. Shipper must include either a Federal Express shipping label identifying the USGS internal cost code or Federal Express account number, or a return mailing label and metered with correct postage for return of the cooler by parcel post. RBPGL is not open on Saturday, Sunday or Federal holidays, therefore, coolers should not be sent the day before a holiday. Any cooler or container not accompanied by a prepaid shipping return label will not be returned automatically. For answers to additional questions sample submitters should email *RBPGL@usgs.gov*.

Sample Preparation

Sample preparation at the RBPGL includes logging samples into LIMS-NVDOCS in batches of 1-60 samples per batch, printing labels for each sample and printing a summary project report. Each

sample label contains the field id (the identifier by which the sample submitter knows each sample) and the unique RBPGL lab id assigned by the LIMS-NVDOCS log in procedure. This information is e-mailed to the submitter of the samples for identification of any errors. The processing of samples involves verifying that samples have been acidified to pH of ≤ 2 , and field id and dates correspond to the list provided by the submitter.

Holding Time, Sample Analysis, and Time Requirements

The maximum time that filtered, acidified, and chilled samples can be held prior to analysis is 28 days. A sample template for as many as 60 samples is created in LIMS-NVDOCS. The analyst sets up blanks, certified reference material, conditioning blanks, duplicate or triplicate samples, calibration standards, and external standards in the template. Each sample is run in duplicate. The standard error for duplicate runs of the same sample should be $\leq 3\%$. Average of the duplicates is the reported value. Average, median, standard deviation (SD), and percent relative standard deviation (%RSD) for duplicate or triplicate samples are reported. The LIMS-NVDOCS will not release preliminary data to submitters until the statistics are acceptable. The time requirement for performing the NVDOC analyses is a minimum of ten days for every 10 samples. If a submitter requires a shorter turnaround time for the analyses, contact RBPGL.

Problematic Samples

Problematic samples can include surface water and groundwater impacted by landfill leachate, brines, industrial and domestic wastewater and hydraulic fracturing or oil exploration sites. Such samples may require additional preparation in order to improve analytical results and/or remove interferences. Samples from landfills which contain substantial amounts of organic matter must be greatly diluted. For samples with high alkalinity, the sparging time must be adjusted to be sure that CO₂

is eliminated, thereby preventing inorganic carbon from interfering with the dissolved organic carbon measurement. Brine samples require more frequent replacement of the halogen scrubber. The salt content of these types of samples do not combust, and so therefore, gradually build a residue inside the combustion tube eventually clogging the catalyst. This residue build up requires more regeneration or replacement of the catalyst. Insufficient dilution, filtration, acidification and poor combustion of samples may lead to instrument interferences that can preclude obtaining acceptable.

Sample Retention Time and Disposal

Water samples are retained by the RBPGL for 30 days after data are reported. Samples are then discarded unless the submitter requests that the samples be returned. Return shipping costs are paid for by the sample submitter. Sample-analysis raw-data files generated from the TOC-Control V system software are exported in Microsoft Excel[®] format and copies are kept for 6 months on two different servers, one on the instrument computer and one on a shared network.

The Analytical System and Measurement of NVDOC, Shimadzu TOC-V csn Total Organic Carbon Analyzer

Equipment

The NVDOC, measured in a sample analyzed on the TOC-V csn Total Organic Carbon Analyzer (referred to herein as TOC-V), is converted to CO₂ by catalytic combustion and measured directly by an NDIR detector, (fig. 1).

The ASI-V autosampler, TOC-V Analyzer, and TOC-Control V System Software are the major components of the analytical system. Together, they meet or exceed all the specifications according to the Standard Methods 5310B, US EPA Methods 415.1, EPA SW-846 Method 9060A, and ASTM 2579 (Shimadzu Instruction Manual, 2001). The injection system on the autosampler uses a μL syringe that is designed to maintain both accurate and precise measurements. The average analytical run time is approximately 5 minutes per injection. Multiple injections of each sample continue until at least three injections meet the system-specified range of a SD of ≤ 0.1 area counts, $\text{CV} \leq 2\%$, or the best three of five injections. The sample tray holds a maximum of 93 24-mL vials. The carrier gas is zero air. HCl is added to the samples. The external sparging kit (an additional sparging sequence allowing for the removal of inorganic carbon outside the sample injector syringe) allows parallel sparging and measurement; as one sample vial is being analyzed, the next sample is in a pretreatment mode (acidified and sparged). The TOC-V uses a NDIR detector for maximum sensitivity and durability, platinum catalyst, and $680\text{ }^{\circ}\text{C}$ combustion to oxidize the water samples. The combustion type NVDOC analysis eliminates most salt interferences and enables complete oxidation (Shimadzu 2001). The auto-dilutor of the TOC-Control V System Software enables the analyst to create a 14-point calibration curve from one standard (fig. 2). For instrument operating instructions and parameters, refer to Appendices 1, 2 and 3.

Reagents, Blanks, Standards, Quality Assurance and Quality Control

Distilled $18.2\ \Omega$ Milli-Q water is used in preparation of reagents, blanks, standards, reference material and dilution of samples. Class A volumetric flasks and pipettes are used to prepare all solutions. All glassware is thoroughly cleaned by soaking for several hours in Alconox[®], (White Plains, NY) and then rinsing one time with tap water and multiple times with $18.2\ \Omega$ water. Foil is used to wrap

the glassware, and non-volumetric items are baked in a muffle furnace for 4 hours at 425 °C. Pipettes are rinsed three times with 18.2 Ω water, and then air-dried. After air-drying over night, they are baked in a muffle furnace for 4 hours at 200 °C, allowed to cool, and then wrapped in foil. Volumetric glassware is dried for 4 hours at 200 °C. Glassware is cooled to room temperature before use. Acidified VFW (with 2 N HCl) is used as blanks for each run. A minimum of eight blanks are run at the beginning of each batch and 4 at the end of each batch. The blanks are run throughout and at the end of the batch to confirm a stable baseline was attained. A median value of the blanks is determined to be the absolute value of the y intercept from peaks integrated with Shimadzu's software (Shimadzu 2001).

Table 1. Batch data with peak areas, concentrations, and calculations for NVDOC analyses of blanks.

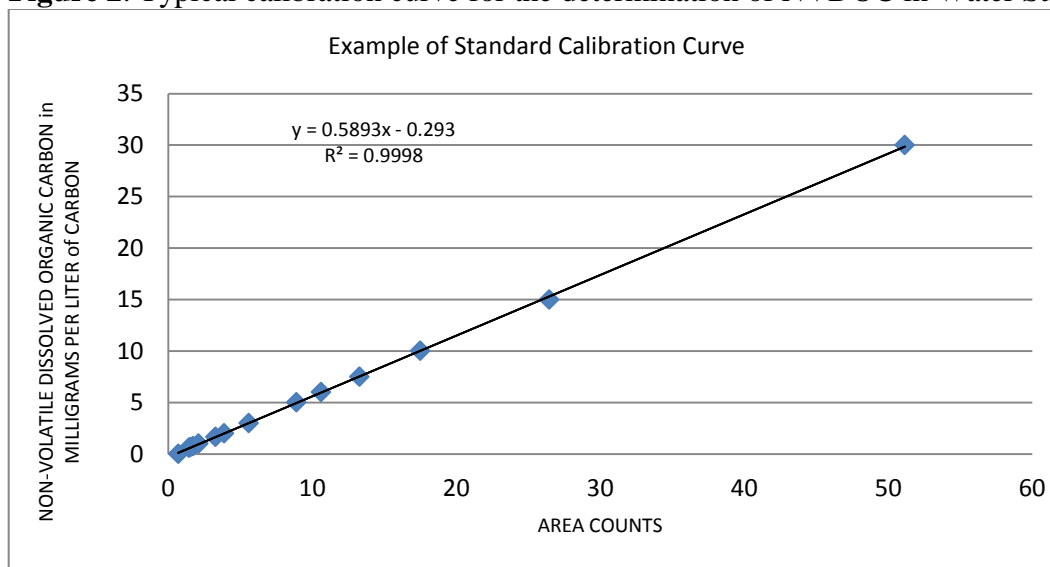
[Standard deviation, SD; Percent relative standard deviation, %RSD]

Blank sample numbers	NVDOC area counts	NVDOC concentrations in mg/L of C	Calculations	Data values
		Analysis		
1	0.690	0.0103		
2	0.710	0.0254		
3	0.720	0.0329		
4	0.692	0.0118		
5	0.730	0.0405	median	0.026
6	0.711	0.0262	average	0.024
7	0.714	0.0284	SD	0.011
8	0.725	0.0367	%RSD	46.0
9	0.690	0.0103		
10	0.700	0.0179		

Standard curves are visually inspected for the presence of outliers or non-linear values. When noted, these are excluded from the calibration curve. Phthalic acid dipotassium salt (99%) [C_6H_4 -1, 2- $(CO_2K)_2$ MW 242.3] (Sigma-Aldrich, St. Louis, MO) is used to make the calibration standard; with two or more external standards also being prepared from the phthalic acid dipotassium salt stock standard. Certified reference material in concentrate form is purchased from Environmental Resource Associates

(ERA, Golden, CO). Concentrations for blanks and standards are calculated from the R^2 equation derived from the standard calibration curve, fig. 2. Equation 2.

Figure 2. Typical calibration curve for the determination of NVDOC in Water Samples.



The NVDOC sample concentration is calculated using the following equation:

$$y = mx + b$$

Where:

y = NVDOC mg/L

m = the slope of the regression line

x = the area count

b = the y-intercept

Preparation of Reagents

2 N HCl

Add 100 mL of 37% Reagent Grade HCl to 500-mL volumetric flask containing approximately 475-mL 18.2 Ω water and fill flask with additional 18.2 Ω water to the fill line. Always

add acid to water to avoid creating a dangerous exothermic reaction. A final concentration accuracy of $\pm 2\%$ is acceptable.

Preparation of Standards

Calibration Standards

Stock Standard Preparation Calculations

(phthalic acid dipotassium salt)

$\text{C}_6\text{H}_4\text{-1,2-(CO}_2\text{K)}_2$ formula weight: 242.32 mg/mmol

Mole percent C $12 \times 8 \div 242.32 = 39.61\%$

Desired Stock Standard: $\text{C}_6\text{H}_4\text{-1,2-(CO}_2\text{K)}_2 = 100 \text{ mg/L C}$

mg $\text{C}_6\text{H}_4\text{-1,2-(CO}_2\text{K)}_2$ needed for 500 mL vol $252.46 \text{ mg} \div 2 = 126.23 \text{ mg } \text{C}_6\text{H}_4\text{-1,2-(CO}_2\text{K)}_2$

Dry approximately 0.50 grams (gm) of phthalic acid dipotassium salt in a drying oven for 30 minutes (min). Cool the salt in a desiccator for 20-30 min. Weigh exactly **0.1262 gm** of the salt using an analytical balance. Add the dried salt to a **500-mL** volumetric flask and fill flask to the fill line with 2 *N* HCl acidified water made previously; this is a 100-mg/L stock standard. Cap the flask and mix well. Dilute 30 mL of the 100-mg/L stock standard with 70 mL of 2 *N* HCl in a 100-mL volumetric flask. Fill flask to the fill line. Cap the flask and mix well. This 30-mg/L high standard is used by the auto-dilutor of the TOC-Control V system software to create a 14-point standard calibration curve. The same procedure is used to make another other dilutions.

Reference Standards

Add approximately 100-200-mL of VFW to a clean 500-mL volumetric flask. Shake the Potable WatR™ Organic Carbon referenced vial prior to opening. Using a clean, dry pipette, volumetrically pipette 5.0 mL of the concentrate into the 500-mL volumetric flask. Fill flask to the fill line volume with

VFW. Cap the flask and mix well. Immediately analyze the diluted sample by normal procedures.

Concentrations of the reference standards are in the mid-calibration range; currently 4-9 mg/L of C.

Reference standards are refrigerated immediately after use. Concentrate holding time under refrigeration is approximately 45 days. Reference standards are discarded if sample run analyses SD are $\geq 3\%$ or older than 45 days.

External Standards

A 5-mg/L external standard is prepared by diluting 5 mL of a 100-mg/L stock standard with 95 mL of 2 *N* HCl in a 100-mL volumetric flask. Fill the flask to the fill line. Cap the flask and mix well. A 10-mg/L external standard is prepared by diluting 10 mL of a 100-mg/L stock standard with 90 mL of 2 *N* HCl in a 100-mL volumetric flask. Fill the flask to the fill line. Cap the flask and mix well. To make a 40 mg/L external standard, dilute 40 mL of the 100-mg/L stock standard with 60 mL 2 *N* HCl in a 100-mL volumetric flask. Fill the flask to the fill line. Cap the flask and mix well. This high standard is run approximately every other run, but is only used to check linearity beyond the standard curve developed using standards 1-14.

Quality Assurance and Quality Control

Field filtered samples are run in duplicate or triplicate with blanks, conditioning blanks, certified reference materials, calibration standards and external standards with every batch. The blanks are run to establish a stable baseline and monitor any contamination during analysis of samples. Conditioning blanks are run before and after the calibration curve. They help in decreasing the instrument blank concentrations by reducing carryover and help restore baseline conditions. Calibration standards are used to calibrate the instrument and the external standards are used to check the accuracy of the calibration standards.

Detection Limits

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. This is determined from analysis of a standard in a given matrix containing the analyte. This procedure is described in 40 CFR part 136, Appendix B, rev. EPA Method 415.1, 1.11 www.caslab.com/EPA-Method-415_1/. The statistical method and data for nine replicate standards being analyzed are listed in table 2. The MDL is calculated using the following equation:

$$MDL = t_{(n-1, 1-\alpha=0.99)}(S)$$

Where:

$MDL = t_{(n-1, 1-\alpha=0.99)}$ = the student's t value appropriate for a 99% confidence level and a standard deviation estimate with $n-1$ degrees of freedom. (The t value for 10 replicates = 2.821),

(S) = standard deviation of the replicate analyses (%).

dnr.wi.gov/regulations/labcert/documents/guidance/-LODguide.pdf

Table 2. Batch Data with Peak Areas, Concentrations, and Calculations for NVDOC Analyses of a 0.5 mg/L Standard.

Standard	NVDOC area counts	NVDOC concentrations in mg/L of C	Calculations	Data values
Analysis				
1	1.41	0.539	Median (mg/L)	0.50
2	1.34	0.495	Average (mg/L)	0.53
3	1.31	0.476	SD(mg/L)	0.03
4	1.33	0.489	%RSD (mg/L)	5.01
5	1.4	0.533		
6	1.29	0.464		
7	1.37	0.514	MDL (mg/L)	0.08
8	1.39	0.527		
9	1.33	0.489		
10	1.4	0.533		

Potential Interferences

Organic particles can cause interferences during sample analysis, yielding a high bias. Proper sample filtration should remove particles in the water samples. Dissolved inorganic carbon is the most significant interference in the measurement of NVDOC. The presence of carbonate and bicarbonate in the sample increases the concentration of CO₂ that is detected if this inorganic carbon is not sufficiently removed prior to sample analysis. The addition of the 2 N HCl, which lowers the pH to 2, converts all the inorganic carbon to CO₂. Subsequent sparging of the samples with Zero air for a predetermined time, removes all this CO₂ before the sample is injected into the combustion tube.

Sample Run

A batch of 60 samples including duplicates or triplicates is run with approximately four External Standards (used for checking calibration) and four Certified Reference Material are analyzed in each run. An analyst examines the raw data and calculates the NVDOC concentrations. Average, median, SD and % RSD are calculated. If the % RSD exceeds 3% for any sample, that sample is re-examined and is added back on the next available day's analytical template.

Data Management and Records

Reported results are sent to submitters. Raw data runs are filed by project and date. Schedule calendars are updated as new submission and sample analyses requests are received.

Health, Safety, and Waste-Disposal Information

Applicable Health and Safety Issues

Personal Protection

Safety glasses, protective gloves, enclosed shoes and lab coats are recommended whenever water samples to be analyzed for NVDOC content are handled, especially when the samples are of biological origin. For other precautions and safety procedures, consult SDS (Safety Data Sheets)

www.ilpi.com/msds/index.html.

Chemical Hazards

HCl is used in preparation of the standards and in the sparging of the samples. Solutions of given HCl concentrations should be made in the fume hood, always adding concentrated acid to water. Soda lime in the CO₂ absorber is caustic and is a known skin irritant. Gloves, eye protection, and liquid-resistant lab coats or aprons should be worn when handling strong acids. Phthalic acid dipotassium salt is an irritant chemical substance, Organic Carbon PotableWatR™ (reference

standard) is a mild reactive and irritant chemical substance, the high sensitivity catalyst (platinum catalyst on quartz wool) is a sensitizer and mild irritant chemical substance, and the halogen scrubber (copper wool) is an irritant chemical substance.

Specific Waste Disposal Requirements

Sample containers must be emptied before disposal. Samples to be discarded should be emptied into a sink containing a trap to neutralize the samples before entering main waste disposal stream. If trap is not available, samples should be neutralized by calcium carbonate or another appropriate buffering agent.

References Cited and Recommended Readings

American Society for Testing and Material, 1991, Standard Specifications for Reagent Water. D1193-77 (Reapproved 1983). Annual Book of ASTM Standards, Vole 11.01. ASTM: Philadelphia, PA.

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dnr.wi.gov/regulations/labcert/documents/guidance/-lodguide.pdf access date 02_12_2014

<http://www.eraqc.com/Resources/ProductCatalogs> access date 02_12_2014

<http://nasl.cbl.umces.edu/Dissolved%20Organic%20Carbon%20sop%202011.pdf> access date 02_12_2014

<http://nwql.usgs.gov/Public/pubs/Method%20holding%20times%205-25-10.pdf> access date 02_12_2014

<http://www.scispec.com/> access date 04_15_2015

<http://www.sigmaaldrich.com/analytical-chromatography/vials/precleaned.html> access date 02_12_2014

https://us.vwr.com/store/catalog/product.jsp?catalog_number=BD309653 access date 04_15_2015

http://www.epa.gov/emap2/html/pubs/docs/resdocs/qa_terms.html access date 02_12_2014

http://www.epa.gov/ogwdw/methods/pdfs/manual_labcertification.pdf access date 02_12_2014

<http://nasl.cbl.umces.edu/Dissolved%20Organic%20Carbon%20sop%202011.pdf> access date 02_12_2014

water.usgs.gov/nrp/biogeochemical-processes-in-groundwater access date 03_10_2015

www.ilpi.com/msds/index.html access date 02_19_2014